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**(54) EMULSION COMPOSITION FOR SILICONE RUBBER SPONGE AND PROCESS FOR PRODUCING SILICONE RUBBER SPONGE**

**(57)Abstract:**

**PROBLEM TO BE SOLVED:** To provide an emulsion composition for a silicone rubber sponge that gives a silicone rubber sponge having uniform and fine cells and having good dimensional precision and to provide a process for producing the silicone rubber sponge using the emulsion composition.

**SOLUTION:** The emulsion composition for the silicone rubber sponge comprises (A) an addition reaction-curable liquid silicone rubber composition comprising (a) a diorganopolysiloxane having at least two alkenyl groups in one molecule, (b) an organopolysiloxane having at least two silicon atom-bonded hydrogen atoms in one molecule and (c) a platinum-based catalyst, (B) water containing a water-soluble polymer and (C) an emulsifier. The composition contains the component (A), the component (B) and the component (C) in an amount of 50-250 pts.wt. of the component (B) and 0.1-10 pts.wt. of the component (C) based on 100 pts.wt. of the total of the component (a), (b) and (c) in the component (A). The process for producing the silicone rubber sponge comprises curing the emulsion composition and then dehydrating.

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**CLAIMS**

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[Claim(s)]

[Claim 1]

(A) Diorganopolysiloxane which has at least two alkenyl groups in a one molecule, (b) Contain an addition reaction hardening type liquid silicone rubber composition containing organopolysiloxane and (c) platinum system catalyst which have at least two silicon atom absorbed water matter in one molecule, water containing B water solubility polymer, and the (C) emulsifier,

An emulsion constituent for silicone rubber sponge characterized by including the above (B) by 50 - 250 weight section, and including the above (C) at a rate of 0.1 - 10 weight section to total quantity 100 weight section of (a) in the above (A), (b), and (c).

[Claim 2]

(B) The emulsion constituent for silicone rubber sponge according to claim 1, wherein loadings of water-soluble polymer to water in an ingredient are 0.1 to 5 % of the weight.

[Claim 3]

(C) The emulsion constituent for silicone rubber sponge according to claim 1, wherein an emulsifier of an ingredient is a nonionic surface active agent.

[Claim 4]

A manufacturing method of silicone rubber sponge hardening the emulsion constituent for silicone rubber sponge according to any one of claims 1 to 3, forming a humid silicone rubber sponge Mr. Plastic solid, removing water from said Plastic solid subsequently, and making with silicone rubber sponge.

[Claim 5]

A manufacturing method of the silicone rubber sponge according to claim 4, wherein an emulsion constituent for silicone rubber sponge before hardening does not contain air bubbles.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]

This invention relates to the manufacturing method of the emulsion for silicone rubber sponge, and silicone rubber sponge.

[0002]

[Description of the Prior Art]

Silicone sponge rubber is excellent in heat resistance and weatherability, and since it is lightweight, it is used taking advantage of this characteristic as a surface coating material; several-kinds sealant of autoparts; copying machine rolls, such as packing, a gasket, and an O ring, etc. Conventionally, as this silicone rubber sponge plasticity constituent, many constituents are proposed as follows.

[0003]

For example, the constituent for silicone rubber sponge which blends the heat decomposition type blowing agent represented by azobisisobutyronitrile is known (for example, the patent documents 1, two references). It blends with silicone rubber by using as a foaming agent composition the emulsion which consists of organopolysiloxane, an emulsifier, water, and a \*\*\*\* agent, and the constituent made to foam using evaporation of water and expansion is proposed (refer to patent documents 3). The silicone rubber sponge composition with which husks blended with the plastic the balloon etc. in which volatile matter like butane or isobutane was included is proposed (refer to patent documents 4). However, since a heat decomposition type blowing agent uses evaporation of gas and water by which it is decomposed and generated, and the steam which expanded as a foaming agent and is using the balloon of thermal expansion nature as a foaming agent at the time of foaming, Although the mold goods of simple shape were turned to, the problem of being unsuitable is among the mold-goods

uses which have complicated shape, and the use was limited.

[0004]

The condensation reaction hardening type silicone emulsion which consists of hydroxy end blockade diorganopolysiloxane and an organic tin catalyst is frozen and hardened, Although the method of removing water and producing silicone rubber sponge after defrosting was known (refer to patent documents 5), many energies were needed for freezing and defrosting, and this method took the long time by Plastic solid completion, and there was a problem that productivity was bad. According to the reaction mechanism which makes alkenyl group content diorganopolysiloxane and the ORGANO hydrogen polysiloxane hydrosilylate under existence of a platinum system catalyst. The constituent which has silicone rubber sponge formed of the hydrogen gas which made water live together and was emitted at the reaction of this ORGANO hydrogen polysiloxane and water when constructing a bridge and acquiring a rubber-like elasticity object (refer to patent documents 6), Or the constituent (refer to patent documents 7) which used liquefied alcohol instead of water is proposed.

[0005]

By the system which stiffens alkenyl group content diorganopolysiloxane by an addition reaction with the ORGANO hydrogen polysiloxane. The silicone rubber sponge composition using water, univalent, or polyhydric alcohol is known, using the ORGANO hydrogen polysiloxane superfluously (refer to patent documents 8). When hardening each these addition reaction hardening type silicone rubber sponge composition, Hydrogen gas and water are used as a foaming agent, and there was a problem that the cellular structure becomes uneven with the material charge to a cavity in compression molding or an injection molding, or a die-components size differed from the part size after shaping, and dimensional accuracy worsened. And the method of manufacturing silicone rubber sponge from the emulsion constituent and emulsion constituent for producing silicone rubber sponge from an addition reaction hardening type silicone emulsion to these patent documents is not indicated.

[0006]

The method of manufacturing the emulsion constituent and silicone rubber sponge for producing silicone rubber sponge from an addition reaction hardening type silicone emulsion composition is proposed (refer to patent documents 9), Here, it was the feature to use absorptivity polymer as a gelling agent for emulsion stabilization, and although it was suitable for the compression-molding use and being excelled in the dimensional accuracy of the sponge Plastic solid which is mold goods, there was a problem that the fineness of a cell and the homogeneity of a cell were missing.

[0007]

[Patent documents 1]

JP,44-461,B (claim)

[Patent documents 2]

JP,7-247436,A (claim 1 and [0011])

[Patent documents 3]

JP,7-122000,B (claim 1, [0063], etc.)

[Patent documents 4]

JP,5-209080,A (claims 1-3)

[Patent documents 5]

JP,59-12832,A (claim)

[Patent documents 6]

JP,54-135865,A (claim 11)

[Patent documents 7]

JP,5-70692,A (claims 1 and 6 etc.)

[Patent documents 8]

JP,11-130963,A (claims 1 and 2)

[Patent documents 9]

JP,2002-114860,A (claims 1-11, [0014], etc.)

[0008]

[Problem(s) to be Solved by the Invention]

The purpose of this invention carries out bridge construction hardening of the emulsion for addition reaction hardening type silicone rubber sponge, After removing water, it has a uniform and fine cell, and it is in providing the manufacturing method of the emulsion constituent for silicone rubber sponge for moreover obtaining silicone rubber sponge with sufficient dimensional accuracy, and silicone rubber sponge.

[0009]

[Means for Solving the Problem]

This invention persons reached this invention, as a result of inquiring that the above-mentioned problem should be canceled. That is, this invention provides a manufacturing method of the following emulsions for silicone rubber sponge, and silicone rubber sponge.

1) Diorganopolysiloxane which has at least two alkenyl groups in (A) a one molecule, (b) Contain an addition reaction hardening type liquid silicone rubber composition containing organopolysiloxane and (c) platinum system catalyst which have at least two silicon atom absorbed water matter in one molecule, water containing B water solubility polymer, and the (C) emulsifier,

An emulsion constituent for silicone rubber sponge characterized by including the above (B) by 50 - 250 weight section, and including the above (C) at a rate of 0.1 - 10 weight section to total quantity 100 weight section of (a) in the above (A), (b), and (c).

[0010]

2) Emulsion constituent for silicone rubber sponge given [ above-mentioned ] in 1 paragraph, wherein loadings of water-soluble polymer to water in the (B) ingredient are 0.1 to 5 % of the weight.

3) Emulsion constituent for silicone rubber sponge given [ above-mentioned ] in 1 paragraph, wherein an emulsifier of the (C) ingredient is a nonionic surface active agent.

4) A manufacturing method of silicone rubber sponge hardening an emulsion constituent for silicone rubber sponge of a statement to either of the one to 3 above-mentioned paragraphs, forming a humid silicone rubber sponge Mr. Plastic solid, removing water from said Plastic solid subsequently, and making with silicone rubber sponge.

[0011]

5) A manufacturing method of silicone rubber sponge given [ above-mentioned ] in 4 paragraphs, wherein an emulsion constituent for silicone rubber sponge before hardening does not contain air bubbles.

[0012]

[Embodiment of the Invention]

The addition reaction hardening type liquid silicone rubber composition of the (A) ingredient used for this invention, Are silicone rubber which presents and hardens the shape of liquid at ordinary temperature, and has rubber-like elasticity, and as this liquid silicone rubber composition, It is an addition reaction hardening type liquid silicone rubber composition which consists of alkenyl group content diorganopolysiloxane and a silicon atom absorbed water matter atom content ORGANO hydrogen polysiloxane, hardens according to a platinum system catalyst and serves as silicone rubber.

[0013]

As this addition reaction hardening type liquid silicone rubber composition, It is a liquid silicone rubber composition which specifically consists of the diorganopolysiloxane which has at least two alkenyl groups in the (a) one molecule, organopolysiloxane which has at least two silicon atom absorbed water matter atoms in one molecule (b), and a (c) platinum system catalyst. This constituent is explained. First, organopolysiloxane of the (a) ingredient has at least two silicon atom joint alkenyl groups in one molecule, and a vinyl group, an allyl group, a propenyl group, etc. are illustrated as an alkenyl group. As organic groups other than an alkenyl group, alkyl halide groups, such as aryl group; 3, such as alkyl group; phenyl group [, such as a methyl group, an ethyl group, and a propyl group, ] and tolyl group, 3, and 3-triphloropropyl group and 3-chloropropyl group, etc. are mentioned. The molecular structure for Motoshige may be any of straight chain shape and straight chain shape including branching. Although the molecular weight in particular for Motoshige is not limited, it is preferred that the viscosity at 25 \*\* is 100 or more mPa-s and 100,000 mPa-s or less. In this invention, two or more sorts of above-mentioned organopolysiloxane may be combined.

[0014]

Next, the addition reaction of the organopolysiloxane of the (b) ingredient is carried out, and the silicon atom absorbed water matter atom for Motoshige constructs a bridge, and makes the silicon atom joint alkenyl group of organopolysiloxane in the (a) ingredient to be a cross linking agent and harden it under existence of the platinum system catalyst of the (c) ingredient. A part for Motoshige needs to have three or more silicon atom absorbed water matter atoms preferably in [ at least two ] one molecule. (b) As a silicon atom joint organic group in an ingredient, alkyl halide groups, such as aryl group;3, such as alkyl group; phenyl group [ , such as a methyl group, an ethyl group, and a propyl group, ] and tolyl group, 3, and 3-triphloropropyl group and 3-chloropropyl group, etc. are mentioned. Any of straight chain shape, the letter of branching, annular, and mesh shape may be sufficient as molecular structure also by Motoshige. Although the molecular weight in particular for Motoshige is not limited, it is preferred that the viscosity at 25 \*\* is 3 - 10,000 mPa-s.

[0015]

(A) The loadings in an ingredient are quantity that the mole ratio of the silicon atom joint alkenyl group in the silicon atom absorbed water matter atom in the (b) ingredient and the (a) ingredient becomes - (0.5:1) (50:1), This is because the hardness of the silicone rubber sponge which is a hardened material will become high too much if larger [ if this mole ratio is smaller than 0.5, good hardenability will not be acquired, but ] than 50. The platinum system catalyst of the (c) ingredient is a catalyst for making it harden by an addition reaction, for example, the olefin complex of platinum impalpable powder, platinum black, chloroplatinic acid, and chloroplatinic acid, a complex compound with the alkenyl siloxane of chloroplatinic acid, a rhodium compound, and a palladium compound are illustrated. The adequate amount as a catalyst is used for these.

[0016]

In order to adjust mobility in this liquid silicone rubber composition or to raise the mechanical strength of a hardened material to it, may blend various kinds of bulking agents with it, and as such a bulking agent, Precipitated silica, fumed silica, carbon black, pyrogenic silica, colloid calcium carbonate, Reinforcement nature bulking agents, such as a fumed titanium dioxide; The end of quartz powder, and silicon ground, aluminosilicate, Un-reinforcing nature bulking agents, such as magnesium oxide and sedimentation method calcium carbonate; dimethyldichlorosilane and the thing which passed and carried out hydrophobing processing with organic silicon compounds, such as KISAME chill disilazane and octamethylcyclotetrasiloxane, are mentioned in the bulking agent of these. Alcohols, paints, an addition reaction depressant, a heat-resistant agent, fire retardant, a plasticizer, an adhesive grant agent, etc. may be blended if needed.

[0017]



The water in the (B) ingredient used for this invention is an ingredient required for production of the emulsion for silicone rubber sponge, if it is pure, it is good, and the kind is not restricted. Tap water, well water, ion exchange water, and distilled water are illustrated. (B) The loadings of an ingredient are 50 per total quantity 100 weight section of (a) in the (A) ingredient, (b), and (c) - 250 weight section, and are 70 - 200 weight section preferably. It is because the expansion ratio of the silicone rubber sponge which will be formed if less than 50 weight sections is small, and the intensity of the silicone rubber sponge formed will be spoiled if 250 weight sections are exceeded. (B) Conventionally, it may be used as the thickener and the viscous agent of an emulsion, the water-soluble polymer contained in an ingredient has the high viscosity of the aqueous thing at the time of dissolving in water, and it is blended in order to prepare a stable emulsion in the amount of low emulsifiers. The loadings to the water are 0.5 to 3 % of the weight preferably 0.1 to 5% of the weight. When the quantity is lower than 0.1 % of the weight, the viscosity of the aqueous thing which water-soluble polymer dissolved is low, and when exceeding 5 % of the weight, viscosity is too high, deaerating from an emulsion becomes [ a stable emulsion is difficult to get, ] difficult, and it is not all desirable.

[0018]

As water-soluble polymer in an ingredient, (B) Alginic acid, sodium alginate, The sodium salt of carboxylate, the sodium salt of carboxymethyl cellulose, Although the sodium salt of methyl cellulose, cellulose ether, hydroxyethyl cellulose, denaturation starch, polyvinyl alcohol, polyacrylate, or polymethacrylate, etc. are illustrated, a higher thing has more preferred viscosity when it dissolves in water.

[0019]

Although it is blended in order that the emulsifier of the (C) ingredient used for this invention may form a stable emulsion, and the kind in particular is not limited, generally a nonionic emulsifier is preferred. The nonionic surface active agent as a nonionic emulsifier, Polyoxyethylene alkyl ether, polyoxyethylene alkylphenyl ether, Ethylene glycol mono- fatty acid ester, propylene glycol mono- fatty acid ester, Sorbitan mono- fatty acid ester, sorbitan trifatty acid ester, polyoxyethylene sorbitan trifatty acid ester, Polyoxyethylene mono- fatty acid ester, polio KISHIECHI range fatty acid ester, polyoxyethylene propylene glycol fatty acid ester, or a POE polyhydric alcohol class is illustrated, and one sort or two sorts or more may be used together. The HLB value of the emulsifier to be used has a preferred thing of 6-14, and it is more preferred to use two or more sorts together. (C) Its weight can be substantially reduced with the viscosity of the (B) ingredient, and the loadings of an ingredient are 0.5 - 7 weight section preferably 0.1 to 10 weight section to total quantity 100 weight section of (a) in the (A) ingredient, (b), and (c). Since the physical property after the heatproof of silicone rubber sponge will fall remarkably if it will be hard to become a stable emulsion for silicone rubber sponge if less than 0.1 weight sections, and ten weight sections are exceeded, neither

is preferred.

[0020]

The emulsion for silicone rubber sponge which comprises this (A) ingredient, the (B) ingredient, and the (C) ingredient can be manufactured, for example by the following methods. The (A) liquid silicone rubber composition of the specified quantity, the water containing (B) water solubility polymer of the specified quantity, and the (C) emulsifier of the specified quantity are fed into a high-speed-stirring mixer, An every place fixed quantity of water and the (C) emulsifier which carries out predetermined time stirring mixing, or contains the (a) ingredient in the (A) ingredient and the (B) water soluble polymer is supplied to a high-speed-stirring mixer, and after carrying out predetermined time stirring mixing, stirring mixing of the (b) ingredient in the (A) ingredient of the specified quantity and the (c) ingredient may be carried out. If it is a homomixer, a paddle type mixer, and a thing from which a stable emulsion is obtained although it passes and a NSHIERU mixer, a gay day spar, a colloid mixer, a vacuum mixing stirring mixer, or a biaxial extrusion machine is illustrated as a mixer used here, it will not be limited in particular.

[0021]

While this emulsion for silicone rubber sponge is involving in air bubbles at the time of mixing, it is after deaeration (for example, a compression-molding metallic mold), ordinary temperature - hardening at the temperature of 50-100 °C preferably, and 120 °C of silicone rubber sponge Mr. Plastic solids of a damp or wet condition being made to form, and, Next, by removing water from the silicone rubber sponge Plastic solid of a damp or wet condition by 150-250 °C secondary heat-treatment, a cell is uniform and, moreover, silicone rubber sponge with fine and sufficient dimensional accuracy can be obtained. The emulsion for silicone rubber can be extruded from a nozzle in the shape of a rod, for example, it can be made to be able to harden in 80-100 °C hot water, hot air drying of it can be carried out, and corded silicone rubber sponge can also be produced.

[0022]

[Example]

Hereafter, an example and a comparative example explain this invention in detail. In the following statements, it means weight % that there is that it is with a part with % about a weight section, respectively, and viscosity shows the value in 25 °C. Each characteristic of the emulsion for silicone rubber sponge and silicone rubber sponge was measured on condition of the following.

- Emulsified state; The state of emulsification was externally judged by viewing after predetermined time stirring mixing.
- Density of sponge; After carrying out shaping hardening of the emulsion for silicone rubber sponge at a sheet shaped, the shaping sheet obtained by carrying out hot air drying was

pierced to the punch of the constant diameter, the weight and thickness of the rubber piece which were pierced were measured, and weight/volume ( $\text{g}/\text{cm}^3$ ) was computed as density.

- Dimensional accuracy; The proportion which broke the shaping sheet thickness (mm) after hot air drying by cure molding sheet thickness (mm) before hot air drying was computed as dimensional accuracy.

- Cell diameter; It observed with the magnifying glass.

[0023]

Examples 1-3

The dimethylpolysiloxane (0.14% of vinyl group content) [(a) Ingredient] 100 copy of viscosity 10,000 mPa-s by which chain both ends were blocked by the dimethylvinyl siloxy group, Five copies of hexamethyldisilazanes and one copy of water were added as 20 copies of fumed silica of specific surface area  $2$ [ of 200 m ]/g, and a silica processing agent, it mixed uniformly with the mixer, heat treatment was performed under 180 °C and decompression for 2 hours, and the liquid liquid silicone rubber base was prepared. To 100 copies of this base-SU. Both-ends trimethylsiloxy group blockade dimethylsiloxane / methylhydrogensiloxane copolymer (0.8% of silicon atom absorbed water matter content) [(b) Ingredient] Platinum complex [ of 0.5 copy, chloroplatinic acid, and divinyl tetramethyl disiloxane ] [ (c) as ingredient] 0.1 copy (0.4% of platinum concentration), and a reaction retardant -- The 3,5-dimethyl- 1 - all [ \*\*\*\*\*- 3 ] -- 0.1 copy was added, it mixed uniformly and the liquid silicone rubber composition was obtained. The water which contains the sodium polyacrylate which is water-soluble polymer in this liquid silicone rubber composition (1.0% of water-soluble polymer content), As a nonionic surface active agent, the polyoxyethylene JIRAU rate of HLB value 6.6, Polyoxyethylene dioleate of HLB value 10.4 with the compounding ratio [compounding ratio to 100 copies of total quantities of (a), (b), and the (c) ingredient in said liquid silicone rubber composition] shown in Table 1. It supplied to the commercial loon textile mixer (made by Pine Bara company), it mixed for 10 minutes at the number of rotations of 9000 rpm, and the emulsion for silicone rubber sponge was prepared, respectively.

[0024]

Next, in order to remove air bubbles from the emulsion for silicone rubber sponge of these air-bubbles mixing, after deaerating with a vacuum pump, it slushed into the 2-mm-thick sheet-shaped metallic mold, and it applied for 10 minutes, and hardened and 90 °C of silicone rubber sponge Mr. shaping sheets of the damp or wet condition were obtained. These shaping sheet was put into 150 °C hot wind type oven, hot air drying was carried out over 1 hour, and sheet shaped silicone rubber sponge was obtained, respectively. The density, dimensional accuracy, and a cell diameter were measured, and it was shown in Table 1. From this result, the silicone rubber sponge by this invention of the homogeneity of a cell is also good, and excellent in dimensional accuracy.

[0025]

## Comparative examples 1-2

Other than water-soluble polymer not being included, although the emulsion for silicone rubber sponge was altogether prepared with the compounding ratio of Table 1 according to the procedure of Examples 1 and 2, a stable emulsion could not be produced and the silicone rubber sponge Mr. Plastic solid of the damp or wet condition was not able to be acquired.

## Comparative example 3

100 copies of liquid silicone rubber compositions of Examples 1-3, 120 copies of water containing absorptivity polymer (acrylate polymer partial sodium salt bridge construction thing by Sanyo Chemical Industries, Ltd.) (absorptivity polymer 2.0%), Seven copies of polyoxyethylene JIRAU rates were applied, it mixed, the emulsion for silicone rubber sponge was prepared, and hardening and the result of having carried out hot air drying and having produced silicone rubber sponge were shown in Table 1 by the method of Examples 1-3.

[0026]

[Table 1]

	実施例1	実施例2	実施例3	比較例1	比較例2	比較例3
液状シリコンゴム組成物 (部)(*)	100	100	100	100	100	100
水溶性ポリマー(1.0%)を 含有する水(部)	120	180	120			
吸水性ポリマー(2.0%)を 含有する水(部)						120
水(部)				60	60	
ポリオキシエチレンジオレエ ート(部)	0.6	0.6	0.6		12	
ポリオキシエチレンジラウレ ート(部)	2.5	2.5		18		7
乳化状態	乳化	乳化	乳化	乳化せず	乳化せず	乳化
密度	0.58	0.48	0.61			0.57
寸法精度	1.0	1.0	0.99			0.99
セル径(mm)	0.1~0.2	0.1~0.2	0.1以下			0.2~0.8

(\*) (a), (b) および (c) の合計量 100 部

[0027]

#### Example 4

Chain both ends to the methylvinyl polysiloxane polymer (0.14% of vinyl group content) [(a) Ingredient] 100 copy of viscosity 40,000 mPa-s blocked by the dimethylvinyl siloxy group. The Aerosil R-972 (made by \*\*\*\* Aerosil) 20 copy which carried out the surface treatment by dimethyldichlorosilane was added, it mixed uniformly, heat treatment was performed at 180 \*\* for 2 hours, and the liquid liquid silicone base was prepared. To 100 copies of this \*\*-SU. Both-ends trimethylsiloxy group blockade dimethylsiloxane / methylhydrogensiloxane copolymer (0.8% of silicon atom absorbed water matter content) [(b) Ingredient] Platinum complex [ of 0.5 copy, chloroplatinic acid, and divinyl tetramethyl disiloxane ] [. (c) as ingredient]0.1 copy (0.4% of platinum concentration), and a reaction retardant -- The 3,5-dimethyl- 1 - all [ \*\*\*\*\*- 3 ] -- 0.1 copy was added, it mixed uniformly and the liquid silicone rubber composition was obtained. To this liquid silicone rubber composition as a compounding ratio to 100 copies of total quantities of the aforementioned (a), (b), and (c), 120 copies of water containing the sodium polyacrylate which is water-soluble polymer (2% of water-soluble polymer content), As a higher alcohol system nonionic surface active agent, the SANNONITSUKU SS-50 (made by Sanyo Chemical Industries, Ltd.) 2.5 copy of HLB10.5, The SANNO nick SS-70 (made by Sanyo Chemical Industries, Ltd.) 2.5 copy of HLB12.8 was measured in the container, and the T.K. gay mixer MARKII2.5 type (made by a special opportunity-ized industrial company) was used, and at the number of rotations of 5000 rpm, it applied for 5 minutes, and mixed, and the emulsion for silicone rubber sponge was prepared.

[0028]

After deaerating this emulsion for silicone rubber sponge with a vacuum pump, it slushed into the 2-mm-thick sheet-shaped metallic mold, and hardened over 15 minutes at 90 \*\*, and the silicone rubber sponge Mr. shaping sheet of the damp or wet condition was obtained. Hot air drying of these shaping sheet was carried out by the same method as Example 1, and silicone rubber sponge was obtained. The density, dimensional accuracy, and a cell diameter were measured. As a result, as for 0.59 and dimensional accuracy, 0.98 and the cell diameter of density were 0.05-0.1 mm.

[0029]

#### Comparative example 4

The result carried out on Example 4 and the conditions except [ all ] using 120 copies of water (0.3% of absorptivity polymer content) made to gel by an acrylate polymer partial sodium salt bridge construction thing as absorptivity polymer instead of water-soluble polymer in Example 4, As for 0.58 and dimensional accuracy, 0.98 and the cell diameter of density were 0.2-1.0

mm.

#### Example 5

To 100 copies of liquid silicone rubber composition DY35-700 A/B (made by Dow Corning Toray Silicone) of a precipitated silica system. 120 copies of water which contains sodium polyacrylate as water-soluble polymer (2% of water-soluble polymer content), As a higher alcohol system nonionic surface active agent, SANNO nick SS-50 2.5 copy, After measured the SANNO nick SS-70 2.5 copy in the container, having used the T.K. homomixer, having applied for 5 minutes, mixing at the number of rotations of 5000 rpm and preparing the emulsion for silicone rubber, by the same method as Example 4, it deaerated and hardened, and hot air drying was carried out and silicone rubber sponge was obtained. As for 0.99 and dimensional accuracy, 0.99 and the cell diameter of density were 0.1 mm or less as a result of measurement.

[0030]

#### [Effect of the Invention]

Including a (A) ingredient - (C) ingredient, the emulsion constituent for silicone rubber sponge of this invention is hardened in the state of an emulsion, and has the feature that the silicone rubber sponge Mr. Plastic solid of a damp or wet condition can be formed. Since the manufacturing method of the silicone rubber sponge of this invention carries out hot air drying of the silicone rubber sponge Mr. Plastic solid of a damp or wet condition and serves as silicone rubber sponge, The flattery nature of die shape is also good, and dimensional accuracy is also good, and it excels in the homogeneity of a cell, and has the feature that silicone rubber sponge with a fine cell can moreover be manufactured efficiently.

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**TECHNICAL FIELD**

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[Field of the Invention]

This invention relates to the manufacturing method of the emulsion for silicone rubber sponge, and silicone rubber sponge.

[0002]

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## PRIOR ART

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### [Description of the Prior Art]

Silicone sponge rubber is excellent in heat resistance and weatherability, and since it is lightweight, it is used taking advantage of this characteristic as a surface coating material; several-kinds sealant of autoparts; copying machine rolls, such as packing, a gasket, and an O ring, etc. Conventionally, as this silicone rubber sponge plasticity constituent, many constituents are proposed as follows.

#### [0003]

For example, the constituent for silicone rubber sponge which blends the heat decomposition type blowing agent represented by azobisisobutyronitrile is known (for example, the patent documents 1, two references). It blends with silicone rubber by using as a foaming agent composition the emulsion which consists of organopolysiloxane, an emulsifier, water, and a \*\*\*\* agent, and the constituent made to foam using evaporation of water and expansion is proposed (refer to patent documents 3). The silicone rubber sponge composition with which husks blended with the plastic the balloon etc. in which volatile matter like butane or isobutane was included is proposed (refer to patent documents 4). However, since a heat decomposition type blowing agent uses evaporation of gas and water by which it is decomposed and generated, and the steam which expanded as a foaming agent and is using the balloon of thermal expansion nature as a foaming agent at the time of foaming, Although the mold goods of simple shape were turned to, the problem of being unsuitable is among the mold-goods uses which have complicated shape, and the use was limited.

#### [0004]

The condensation reaction hardening type silicone emulsion which consists of hydroxy end blockade diorganopolysiloxane and an organic tin catalyst is frozen and hardened, Although the method of removing water and producing silicone rubber sponge after defrosting was known (refer to patent documents 5), many energies were needed for freezing and defrosting,



and this method took the long time by Plastic solid completion, and there was a problem that productivity was bad. According to the reaction mechanism which makes alkenyl group content diorganopolysiloxane and the ORGANO hydrogen polysiloxane hydrosilylate under existence of a platinum system catalyst. The constituent which has silicone rubber sponge formed of the hydrogen gas which made water live together and was emitted at the reaction of this ORGANO hydrogen polysiloxane and water when constructing a bridge and acquiring a rubber-like elasticity object (refer to patent documents 6), Or the constituent (refer to patent documents 7) which used liquefied alcohol instead of water is proposed.

[0005]

By the system which stiffens alkenyl group content diorganopolysiloxane by an addition reaction with the ORGANO hydrogen polysiloxane. The silicone rubber sponge composition using water, univalent, or polyhydric alcohol is known, using the ORGANO hydrogen polysiloxane superfluously (refer to patent documents 8). When hardening each these addition reaction hardening type silicone rubber sponge composition, Hydrogen gas and water are used as a foaming agent, and there was a problem that the cellular structure becomes uneven with the material charge to a cavity in compression molding or an injection molding, or a die-components size differed from the part size after shaping, and dimensional accuracy worsened. And the method of manufacturing silicone rubber sponge from the emulsion constituent and emulsion constituent for producing silicone rubber sponge from an addition reaction hardening type silicone emulsion to these patent documents is not indicated.

[0006]

The method of manufacturing the emulsion constituent and silicone rubber sponge for producing silicone rubber sponge from an addition reaction hardening type silicone emulsion composition is proposed (refer to patent documents 9), Here, it was the feature to use absorptivity polymer as a gelling agent for emulsion stabilization, and although it was suitable for the compression-molding use and being excelled in the dimensional accuracy of the sponge Plastic solid which is mold goods, there was a problem that the fineness of a cell and the homogeneity of a cell were missing.

[0007]

[Patent documents 1]

JP,44-461,B (claim)

[Patent documents 2]

JP,7-247436,A (claim 1 and [0011])

[Patent documents 3]

JP,7-122000,B (claim 1, [0063], etc.)

[Patent documents 4]

JP,5-209080,A (claims 1-3)

[Patent documents 5]

JP,59-12832,A (claim)

[Patent documents 6]

JP,54-135865,A (claim 11)

[Patent documents 7]

JP,5-70692,A (claims 1 and 6 etc.)

[Patent documents 8]

JP,11-130963,A (claims 1 and 2)

[Patent documents 9]

JP,2002-114860,A (claims 1-11, [0014], etc.)

[0008]

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## EFFECT OF THE INVENTION

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[Effect of the Invention]

Including a (A) ingredient - (C) ingredient, the emulsion constituent for silicone rubber sponge of this invention is hardened in the state of an emulsion, and has the feature that the silicone rubber sponge Mr. Plastic solid of a damp or wet condition can be formed. Since the manufacturing method of the silicone rubber sponge of this invention carries out hot air drying of the silicone rubber sponge Mr. Plastic solid of a damp or wet condition and serves as silicone rubber sponge, The flattery nature of die shape is also good, and dimensional accuracy is also good, and it excels in the homogeneity of a cell, and has the feature that silicone rubber sponge with a fine cell can moreover be manufactured efficiently.

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**TECHNICAL PROBLEM**

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[Problem(s) to be Solved by the Invention]

The purpose of this invention carries out bridge construction hardening of the emulsion for addition reaction hardening type silicone rubber sponge, After removing water, it has a uniform and fine cell, and it is in providing the manufacturing method of the emulsion constituent for silicone rubber sponge for moreover obtaining silicone rubber sponge with sufficient dimensional accuracy, and silicone rubber sponge.

[0009]

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## EXAMPLE

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[Example]

Hereafter, an example and a comparative example explain this invention in detail. In the following statements, it means weight % that there is that it is with a part with % about a weight section, respectively, and viscosity shows the value in 25 \*\*. Each characteristic of the emulsion for silicone rubber sponge and silicone rubber sponge was measured on condition of the following.

- Emulsified state; The state of emulsification was externally judged by viewing after predetermined time stirring mixing.
- Density of sponge; After carrying out shaping hardening of the emulsion for silicone rubber sponge at a sheet shaped, the shaping sheet obtained by carrying out hot air drying was pierced to the punch of the constant diameter, the weight and thickness of the rubber piece which were pierced were measured, and weight/volume ( $\text{g}/\text{cm}^3$ ) was computed as density.
- Dimensional accuracy; The proportion which broke the shaping sheet thickness (mm) after hot air drying by cure molding sheet thickness (mm) before hot air drying was computed as dimensional accuracy.
- Cell diameter; It observed with the magnifying glass.

[0023]

Examples 1-3

The dimethylpolysiloxane (0.14% of vinyl group content) [(a) Ingredient] 100 copy of viscosity 10,000 mPa-s by which chain both ends were blocked by the dimethylvinyl siloxy group, Five copies of hexamethyldisilazanes and one copy of water were added as 20 copies of fumed silica of specific surface area <sup>2</sup>[ of 200 m ]/g, and a silica processing agent, it mixed uniformly with the mixer, heat treatment was performed under 180 \*\* and decompression for 2 hours, and the liquid liquid silicone rubber base was prepared. To 100 copies of this \*\*-SU. Both-ends

trimethylsiloxy group blockade dimethylsiloxane / methylhydrogensiloxane copolymer (0.8% of silicon atom absorbed water matter content) [(b) Ingredient] Platinum complex [ of 0.5 copy, chloroplatinic acid, and divinyl tetramethyl disiloxane ] [. (c) as ingredient] 0.1 copy (0.4% of platinum concentration), and a reaction retardant -- The 3,5-dimethyl- 1 - all [ \*\*\*\*\*- 3 ] -- 0.1 copy was added, it mixed uniformly and the liquid silicone rubber composition was obtained. The water which contains the sodium polyacrylate which is water-soluble polymer in this liquid silicone rubber composition (1.0% of water-soluble polymer content), As a nonionic surface active agent, the polyoxyethylene JIRAU rate of HLB value 6.6, Polyoxyethylene dioleate of HLB value 10.4 with the compounding ratio [compounding ratio to 100 copies of total quantities of (a), (b), and the (c) ingredient in said liquid silicone rubber composition] shown in Table 1. It supplied to the commercial loon textile mixer (made by Pine Bara company), it mixed for 10 minutes at the number of rotations of 9000 rpm, and the emulsion for silicone rubber sponge was prepared, respectively.

[0024]

Next, in order to remove air bubbles from the emulsion for silicone rubber sponge of these air-bubbles mixing, after deaerating with a vacuum pump, it slushed into the 2-mm-thick sheet-shaped metallic mold, and it applied for 10 minutes, and hardened and 90 \*\* of silicone rubber sponge Mr. shaping sheets of the damp or wet condition were obtained. These shaping sheet was put into 150 \*\* hot wind type oven, hot air drying was carried out over 1 hour, and sheet shaped silicone rubber sponge was obtained, respectively. The density, dimensional accuracy, and a cell diameter were measured, and it was shown in Table 1. From this result, the silicone rubber sponge by this invention of the homogeneity of a cell is also good, and excellent in dimensional accuracy.

[0025]

#### Comparative examples 1-2

Other than water-soluble polymer not being included, although the emulsion for silicone rubber sponge was altogether prepared with the compounding ratio of Table 1 according to the procedure of Examples 1 and 2, a stable emulsion could not be produced and the silicone rubber sponge Mr. Plastic solid of the damp or wet condition was not able to be acquired.

#### Comparative example 3

100 copies of liquid silicone rubber compositions of Examples 1-3, 120 copies of water containing absorptivity polymer (acrylate polymer partial sodium salt bridge construction thing by Sanyo Chemical Industries, Ltd.) (absorptivity polymer 2.0%), Seven copies of polyoxyethylene JIRAU rates were applied, it mixed, the emulsion for silicone rubber sponge was prepared, and hardening and the result of having carried out hot air drying and having produced silicone rubber sponge were shown in Table 1 by the method of Examples 1-3.

[0026]

[Table 1]

	実施例1	実施例2	実施例3	比較例1	比較例2	比較例3
液状シリコンゴム組成物 (部)(*)	100	100	100	100	100	100
水溶性ポリマー(1.0%)を 含有する水(部)	120	180	120			
吸水性ポリマー(2.0%)を 含有する水(部)						120
水(部)				60	60	
ポリオキシエチレンジオレエ ート(部)	0.6	0.6	0.6		12	
ポリオキシエチレンジラウレ ート(部)	2.5	2.5		18		7
乳化状態	乳化	乳化	乳化	乳化せず	乳化せず	乳化
密度	0.58	0.48	0.61			0.57
寸法精度	1.0	1.0	0.99			0.99
セル径(mm)	0.1~0.2	0.1~0.2	0.1以下			0.2~0.8

(\*) (a), (b) および (c) の合計量 100 部

[0027]

#### Example 4

Chain both ends to the methylvinyl polysiloxane polymer (0.14% of vinyl group content) [(a) Ingredient] 100 copy of viscosity 40,000 mPa-s blocked by the dimethylvinyl siloxy group. The Aerosil R-972 (made by \*\*\*\* Aerosil) 20 copy which carried out the surface treatment by dimethyldichlorosilane was added, it mixed uniformly, heat treatment was performed at 180 \*\* for 2 hours, and the liquid liquid silicone base was prepared. To 100 copies of this \*\*-SU. Both-ends trimethylsiloxy group blockade dimethylsiloxane / methylhydrogensiloxane copolymer (0.8% of silicon atom absorbed water matter content) [(b) Ingredient] Platinum complex [ of 0.5 copy, chloroplatinic acid, and divinyl tetramethyl disiloxane ] [ (c) as Ingredient] 0.1 copy (0.4% of platinum concentration), and a reaction retardant -- The 3,5-dimethyl- 1 - all [ \*\*\*\*\*- 3 ] -- 0.1 copy was added, it mixed uniformly and the liquid silicone rubber composition was obtained.

To this liquid silicone rubber composition as a compounding ratio to 100 copies of total quantities of the aforementioned (a), (b), and (c), 120 copies of water containing the sodium polyacrylate which is water-soluble polymer (2% of water-soluble polymer content), As a higher alcohol system nonionic surface active agent, the SANNONITSUKU SS-50 (made by Sanyo Chemical Industries, Ltd.) 2.5 copy of HLB10.5, The SANNO nick SS-70 (made by Sanyo Chemical Industries, Ltd.) 2.5 copy of HLB12.8 was measured in the container, and the T.K. gay mixer MARKII2.5 type (made by a special opportunity-ized industrial company) was used, and at the number of rotations of 5000 rpm, it applied for 5 minutes, and mixed, and the emulsion for silicone rubber sponge was prepared.

[0028]

After deaerating this emulsion for silicone rubber sponge with a vacuum pump, it slushed into the 2-mm-thick sheet-shaped metallic mold, and hardened over 15 minutes at 90 \*\*, and the silicone rubber sponge Mr. shaping sheet of the damp or wet condition was obtained. Hot air drying of these shaping sheet was carried out by the same method as Example 1, and silicone rubber sponge was obtained. The density, dimensional accuracy, and a cell diameter were measured. As a result, as for 0.59 and dimensional accuracy, 0.98 and the cell diameter of density were 0.05-0.1 mm.

[0029]

#### Comparative example 4

The result carried out on Example 4 and the conditions except [ all ] using 120 copies of water (0.3% of absorptivity polymer content) made to gel by an acrylate polymer partial sodium salt bridge construction thing as absorptivity polymer instead of water-soluble polymer in Example 4, As for 0.58 and dimensional accuracy, 0.98 and the cell diameter of density were 0.2-1.0 mm.

#### Example 5

To 100 copies of liquid silicone rubber composition DY35-700 A/B (made by Dow Corning Toray Silicone) of a precipitated silica system. 120 copies of water which contains sodium polyacrylate as water-soluble polymer (2% of water-soluble polymer content), As a higher alcohol system nonionic surface active agent, SANNO nick SS-50 2.5 copy, After measured the SANNO nick SS-70 2.5 copy in the container, having used the T.K. homomixer, having applied for 5 minutes, mixing at the number of rotations of 5000 rpm and preparing the emulsion for silicone rubber, by the same method as Example 4, it deaerated and hardened, and hot air drying was carried out and silicone rubber sponge was obtained. As for 0.99 and dimensional accuracy, 0.99 and the cell diameter of density were 0.1 mm or less as a result of measurement.

[0030]



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## WRITTEN AMENDMENT

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[Written amendment]

[Filing date] January 28 (2004.1.28), Heisei 16

[Amendment 1]

[Document to be Amended] Specification

[Item(s) to be Amended] 0027

[Method of Amendment] Change

[The contents of amendment]

[0027]

### Example 4

Chain both ends to the methylvinyl polysiloxane polymer (0.14% of vinyl group content) [(a) Ingredient] 100 copy of viscosity 40,000 mPa-s blocked by the dimethylvinyl siloxy group. The Aerosil R-972 (made by Japanese Aerosil) 20 copy which carried out the surface treatment by dimethyldichlorosilane was added, it mixed uniformly, heat treatment was performed at 180 \*\* for 2 hours, and the liquid liquid silicone base was prepared. To 100 copies of this \*\*-SU. Both-ends trimethylsiloxy group blockade dimethylsiloxane / methylhydrogensiloxane copolymer (0.8% of silicon atom absorbed water matter content) [(b) Ingredient] Platinum complex [ of 0.5 copy, chloroplatinic acid, and divinyl tetramethyl disiloxane ] [. (c) as ingredient] 0.1 copy (0.4% of platinum concentration), and a reaction retardant -- The 3,5-dimethyl- 1 - all [ \*\*\*\*\*- 3 ] -- 0.1 copy was added, it mixed uniformly and the liquid silicone rubber composition was obtained. To this liquid silicone rubber composition as a compounding ratio to 100 copies of total quantities of the aforementioned (a), (b), and (c), 120 copies of water containing the sodium polyacrylate which is water-soluble polymer (2% of water-soluble polymer content), As a higher alcohol system nonionic surface active agent, the SANNONITSUKU SS-50 (made by Sanyo Chemical Industries, Ltd.) 2.5 copy of HLB10.5, The SANNO nick SS-70 (made by Sanyo Chemical Industries, Ltd.) 2.5 copy of HLB12.8 was measured in the container, and the T.K.

gay mixer MARKII2.5 type (made by a special opportunity-ized industrial company) was used, and at the number of rotations of 5000 rpm, it applied for 5 minutes, and mixed, and the emulsion for silicone rubber sponge was prepared.

[Amendment 2]

[Document to be Amended]Specification

[Item(s) to be Amended]0029

[Method of Amendment]Change

[The contents of amendment]

[0029]

#### Comparative example 4

The result carried out on Example 4 and the conditions except [ all ] using 120 copies of water (0.3% of absorptivity polymer content) made to gel by an acrylate polymer partial sodium salt bridge construction thing as absorptivity polymer instead of water-soluble polymer in Example 4, As for 0.58 and dimensional accuracy, 0.98 and the cell diameter of density were 0.2-1.0 mm.

#### Example 5

To 100 copies of liquid silicone rubber composition DY35-700 A/B (made by Dow Corning Toray Silicone) of a precipitated silica system. 120 copies of water which contains sodium polyacrylate as water-soluble polymer (2% of water-soluble polymer content), As a higher alcohol system nonionic surface active agent, SANNO nick SS-50 2.5 copy, After measured the SANNO nick SS-70 2.5 copy in the container, having used the T.K. gay mixer, having applied for 5 minutes, mixing at the number of rotations of 5000 rpm and preparing the emulsion for silicone rubber, by the same method as Example 4, it deaerated and hardened, and hot air drying was carried out and silicone rubber sponge was obtained. As for 0.59 and dimensional accuracy, 0.99 and the cell diameter of density were 0.1 mm or less as a result of measurement.

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最終頁に続く

(54) 【発明の名称】 シリコーンゴムスポンジ用エマルション組成物およびシリコーンゴムスポンジの製造方法

## (57) 【要約】

【課題】均一で細かなセルを有し、寸法精度のよいシリコーンゴムスポンジを得るためのシリコーンゴムスポンジ用エマルション組成物と、それを用いる前記スポンジの製造方法を提供する。

【解決手段】(A) (a) 1分子中に少なくとも2個のアルケニル基を有するジオルガノポリシロキサン、(b) 1分子中に少なくとも2個のケイ素原子結合水素を有するオルガノポリシロキサンおよび(c) 白金系触媒を含む付加反応硬化型液状シリコーンゴム組成物、(B) 水溶性ポリマーを含有する水、および(C) 乳化剤を含有し、前記(A)における(a)、(b)および(c)の合計量100重量部、前記(B)50〜250重量部、前記(C)0.1〜10重量部の割合で含むシリコーンゴムスポンジ用エマルション組成物、およびそれを硬化後、脱水するシリコーンゴムスポンジの製造方法。

【選択図】 なし

## 【特許請求の範囲】

## 【請求項1】

(A) (a) 1分子中に少なくとも2個のアルケニル基を有するシオルガノポリシロキサン、(b) 1分子中に少なくとも2個のケイ素原子結合水素を有するオルガノポリシロキサンおよび(c) 白金系触媒を含有してなる付加反応硬化型液状シリコンゴム組成物、B) 水溶性ポリマーを含有する水、および(C) 乳化剤を含有し、前記(A)における(a)、(b)および(c)の合計量100重量部に対し、前記(B)を50～250重量部、前記(C)を0.1～10重量部の割合で含むことを特徴とするシリコンゴムスポンジ用エマルジョン組成物。

## 【請求項2】

(B) 成分中の水に対する水溶性ポリマーの配合量が0.1～5重量%であることを特徴とする請求項1記載のシリコンゴムスポンジ用エマルジョン組成物。

## 【請求項3】

(C) 成分の乳化剤が非イオン界面活性剤であることを特徴とする請求項1記載のシリコンゴムスポンジ用エマルジョン組成物。

## 【請求項4】

請求項1～3のいずれかに記載のシリコンゴムスポンジ用エマルジョン組成物を硬化して湿潤のシリコンゴムスポンジ機成形体を形成し、次いで前記成形体から水を除去してシリコンゴムスポンジとなすことを特徴とするシリコンゴムスポンジの製造方法。

## 【請求項5】

硬化する前のシリコンゴムスポンジ用エマルジョン組成物が気泡を含まないことを特徴とする請求項4記載のシリコンゴムスポンジの製造方法。

## 【発明の詳細な説明】

## 【0001】

## 【発明の属する技術分野】

本発明はシリコンゴムスポンジ用エマルジョンとシリコンゴムスポンジの製造方法に関する。

## 【0002】

## 【従来の技術】

シリコンスポンジゴムは、耐熱性、耐候性に優れ、軽量であることから、かかる特性を活かして、パッキング、ガスケット、Oリング等の自動車部品；複写機ロールの表面被覆材；各種シール材などとして使用されている。従来、かかるシリコンゴムスポンジ形成性組成物としては、以下のとおり数多くの組成物が提案されている。

## 【0003】

例えば、アソビスイソブチロニトリルに代表される熱分解型発泡剤を配合してなるシリコンゴムスポンジ用組成物が知られている（例えば、特許文献1、2参照）。オルガノポリシロキサン、乳化剤、水、粘着剤からなるエマルジョンを発泡剤組成物としてシリコンゴムに配合し、水の蒸発、膨張を利用して発泡させる組成物が提案されている（特許文献3参照）。また、殻がフラスチックで、アタンやイソアタンのような揮発性物質が内包されたバルーン等を配合したシリコンゴムスポンジ組成物が提案されている（特許文献4参照）。しかし、発泡時に熱分解型発泡剤が分解して発生するガスや水の蒸発、膨張した水蒸気を発泡剤としたり、また、熱膨張性のバルーンを発泡剤としたりしているために、単純形状の成形品には向いているものの、複雑な形状を有する成形品用途には不向きという問題点があり、その用途は限定されていた。

## 【0004】

また、ヒドロキシ末端封鎖シオルガノポリシロキサンと有機錫触媒からなる縮合反応硬化型のシリコンエマルジョンを凍結して硬化し、解凍後に水を除去してシリコンゴムスポンジを作製する方法が知られているが（特許文献5参照）、この方法では凍結、解凍に多くのエネルギーを必要とし、また成形体完成までに長時間を要し、生産性が低いという問題点があった。さらに、アルケニル基含有シオルガノポリシロキサンとオルガノハイド

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ロジエンポリシロキサンとを白金系触媒の存在下にヒドロシリル化させる反応機構により、架橋してゴム状弾性体を得る際に、水を共存させて、該オルガノハイドロジエンポリシロキサンと水との反応で発生した水素ガスによってシリコーンゴムスポンジを形成される組成物（特許文献6参照）、あるいは水の代りに液状アルコールを用いた組成物（特許文献7参照）が提案されている。

#### 【0005】

さらには、アルケニル基含有ジオルガノポリシロキサンを、オルガノハイドロジエンポリシロキサンとの付加反応によって硬化させる系で、オルガノハイドロジエンポリシロキサンを過剰に用い、かつ水と1価または多価アルコールを用いるシリコーンゴムスポンジ組成物が知られている（特許文献8参照）。これら付加反応硬化型シリコーンゴムスポンジ組成物はいずれも硬化する際に、水素ガスならびに水を発泡剤とするものであり、圧縮成形やインジェクション成形においてはキャビティへの材料仕込み量によってセル構造が不均一になったり、金型の部品寸法と成形後の部品寸法が異なり寸法精度が悪くなったりするという問題点があった。しかもこれら特許文献には付加反応硬化型シリコーンエマルジョンからシリコーンゴムスポンジを作製するためのエマルジョン組成物およびエマルジョン組成物からシリコーンゴムスポンジを製造する方法は記載されていない。

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#### 【0006】

また、付加反応硬化型シリコーンエマルジョン組成物からシリコーンゴムスポンジを作製するためのエマルジョン組成物およびシリコーンゴムスポンジを製造する方法が提案されており（特許文献9参照）、ここではエマルジョン安定化のために、ゲル化剤として吸水性ポリマーを使用しているのが特徴で、圧縮成形用途に好適で、成形品であるスポンジ成形体の寸法精度に優れるものの、セルの細かさやセルの均一性に欠けるという問題点があった。

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#### 【0007】

##### 【特許文献1】

特公昭44-461号公報（特許請求の範囲）

##### 【特許文献2】

特開平7-247486号公報（請求項1および【0011】）

##### 【特許文献3】

特公平7-122000号公報（請求項1、【0063】など）

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##### 【特許文献4】

特開平5-209080号公報（請求項1〜3）

##### 【特許文献5】

特開昭59-12882号公報（特許請求の範囲）

##### 【特許文献6】

特開昭54-136865号公報（請求項11）

##### 【特許文献7】

特開平5-70692号公報（請求項1、6など）

##### 【特許文献8】

特開平11-130968号公報（請求項1、2）

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##### 【特許文献9】

特開2002-114860号公報（請求項1〜11、【0014】など）

#### 【0008】

##### 【発明が解決しようとする課題】

本発明の目的は、付加反応硬化型シリコーンゴムスポンジ用エマルジョンを架橋硬化し、水を除去した後均一かつ細かなセルを有し、しかも寸法精度のよいシリコーンゴムスポンジを得るためのシリコーンゴムスポンジ用エマルジョン組成物とシリコーンゴムスポンジの製造方法を提供することにある。

#### 【0009】

##### 【課題を解決するための手段】

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本発明者らは上記問題点を解消すべく研究した結果、本発明に到達した。すなわち、本発明は、以下のシリコーンゴムスポンジ用エマルジョンとシリコーンゴムスポンジの製造方法を提供するものである。

1) (A) (a) 1分子中に少なくとも2個のアルケニル基を有するジオルガノポリシロキサン、(b) 1分子中に少なくとも2個のケイ素原子結合水素を有するオルガノポリシロキサンおよび(c) 白金系触媒を含有してなる付加反応硬化型液状シリコーンゴム組成物、B) 水溶性ポリマーを含有する水、および(C) 乳化剤を含有し、前記(A)における(a)、(b)および(c)の合計量100重量部に対し、前記(B)を50~250重量部、前記(C)を0.1~10重量部の割合で含むことを特徴とするシリコーンゴムスポンジ用エマルジョン組成物。

【0010】

2) (B)成分中の水に対する水溶性ポリマーの配合量が0.1~5重量%であることを特徴とする上記1)項記載のシリコーンゴムスポンジ用エマルジョン組成物。

3) (C)成分の乳化剤が非イオン界面活性剤であることを特徴とする上記1)項記載のシリコーンゴムスポンジ用エマルジョン組成物。

4) 上記1)~3)項のいずれかに記載のシリコーンゴムスポンジ用エマルジョン組成物を硬化して湿潤のシリコーンゴムスポンジ様成形体を形成し、次いで前記成形体から水を除去してシリコーンゴムスポンジとなすことを特徴とするシリコーンゴムスポンジの製造方法。

【0011】

5) 硬化する前のシリコーンゴムスポンジ用エマルジョン組成物が気泡を含まないことを特徴とする上記4)項記載のシリコーンゴムスポンジの製造方法。

【0012】

【発明の実施の形態】

本発明に使用される(A)成分の付加反応硬化型液状シリコーンゴム組成物は、常温にて液状を呈し、硬化してゴム状弾性を有するシリコーンゴムであり、かかる液状シリコーンゴム組成物としては、アルケニル基含有ジオルガノポリシロキサンとケイ素原子結合水素原子含有オルガノハイドロジェンポリシロキサンとからなり、白金系触媒により硬化してシリコーンゴムとなる付加反応硬化型液状シリコーンゴム組成物である。

【0013】

かかる付加反応硬化型液状シリコーンゴム組成物としては、具体的には、(a) 1分子中に少なくとも2個のアルケニル基を有するジオルガノポリシロキサン、(b) 1分子中に少なくとも2個のケイ素原子結合水素原子を有するオルガノポリシロキサンおよび(c) 白金系触媒からなる液状シリコーンゴム組成物である。

この組成物について説明する。まず、(a)成分のオルガノポリシロキサンは1分子中に少なくとも2個のケイ素原子結合アルケニル基を有しており、アルケニル基としてはビニル基、アリル基、アロペニル基などが例示される。また、アルケニル基以外の有機基としては、メチル基、エチル基、プロピル基などのアルキル基；フェニル基、トリル基などのアリール基；3,3,3-トリフルオロプロピル基、3-クロロプロピル基などのハロゲン化アルキル基などが挙げられる。本成分の分子構造は直鎖状、分枝を含む直鎖状のいずれであってもよい。本成分の分子量は特に限定されないが、25℃における粘度が100 mPa・S以上、100,000 mPa・S以下であることが好ましい。本発明においては上記オルガノポリシロキサンを2種以上組み合わせてもよい。

【0014】

次に、(b)成分のオルガノポリシロキサンは架橋剤であり、(c)成分の白金系触媒の存在下に本成分のケイ素原子結合水素原子が、(a)成分中のオルガノポリシロキサンのケイ素原子結合アルケニル基に付加反応して架橋、硬化させるものである。本成分は1分子中に少なくとも2個、好ましくは8個以上のケイ素原子結合水素原子を有することが必要である。(b)成分中のケイ素原子結合有機基としては、メチル基、エチル基、プロピル基などのアルキル基；フェニル基、トリル基などのアリール基；3,3,3-トリフル

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ロアロビル基、3-クロロロアロビル基などのハロゲン化アルキル基などが挙げられる。本成分も分子構造は、直鎖状、分枝状、環状、網目状のいずれでもよい。本成分の分子量は特に限定されないが、25℃における粘度が3~10,000 mPa・Sであることが好ましい。

#### 【0015】

(A)成分中の配合量は、(b)成分中のケイ素原子結合水素原子と(a)成分中のケイ素原子結合アルケニル基のモル比が(0.5:1)~(50:1)となるような量であり、これは、このモル比が0.5より小さいと良好な硬化性が得られず、50より大きいと硬化物であるシリコンゴムスポンジの硬度が高くなり過ぎるからである。また、(c)成分の白金系触媒は付加反応により硬化させるための触媒であり、例えば、白金微粉末、白金黒、塩化白金酸、塩化白金酸のオレフィン錯体、塩化白金酸のアルケニルシロキサンとの錯化合物、ロジウム化合物、パラジウム化合物が例示される。これらは、触媒としての適当量を用いる。

#### 【0016】

この液状シリコンゴム組成物には、流動性を調節したり、硬化物の機械強度を向上させるために各種の充填剤を配合してもよく、このような充填剤としては、沈降シリカ、ヒュームドシリカ、カーボンブラック、焼成シリカ、コロイド状炭酸カルシウム、ヒュームド二酸化チタンなどの補強性充填剤；石英粉末、珪素土、アルミノケイ酸、酸化マグネシウム、沈降法炭酸カルシウムなどの非補強性充填剤；これらの充填剤をジメチルシクロシラン、ヘキサメチルジシラサン、オクタメチルシクロテトラシロキサンなどの有機ケイ素化合物で疎水化処理したものが挙げられる。さらに、必要に応じてアルコール類、顔料、付加反応抑制剤、耐熱剤、難燃剤、可塑剤、接着性付与剤などを配合してもよい。

#### 【0017】

本発明に使用される(B)成分中の水は、シリコンゴムスポンジ用エマルジョンの作製に必要な成分であり、清浄であればよく、その種類は制限されない。水道水、井戸水、イオン交換水、蒸留水が例示される。(B)成分の配合量は、(A)成分における(a)、(b)および(c)の合計量100重量部当たり50~250重量部であり、好ましくは70~200重量部である。50重量部より少ないと形成されるシリコンゴムスポンジの発泡倍率が小さく、250重量部を超えると、形成されるシリコンゴムスポンジの強度が損なわれるからである。(B)成分中に含有する水溶性ポリマーは、従来、エマルジョンの増粘剤や粘剤として使用されるものでよく、水に溶解した際の水性物の粘性が高く、低乳化剤量で安定なエマルジョンを調製するために配合される。その水に対する配合量は0.1~5重量%、好ましくは0.5~8重量%である。その量が0.1重量%より低いときは水溶性ポリマーが溶解した水性物の粘性が低く、安定なエマルジョンが得難く、5重量%を超えると粘性が高過ぎてエマルジョンからの脱気が困難となり、いずれも好ましくない。

#### 【0018】

(B)成分中の水溶性ポリマーとしては、アルギン酸、アルギン酸ナトリウム、カルボキシレートのナトリウム塩、カルボキシメチルセルロースのナトリウム塩、メチルセルロース、セルロースエーテル、ヒドロキシエチルセルロース、変性デンプン、ポリビニルアルコール、ポリアクリレートあるいはポリメタクリレートのナトリウム塩などが例示されるが、水に溶解したときの粘性が高いものほど好ましい。

#### 【0019】

本発明に使用される(C)成分の乳化剤は、安定なエマルジョンを形成するために配合され、その種類は特に限定されないが、一般に非イオン性の乳化剤が好ましい。非イオン性の乳化剤としての非イオン界面活性剤は、ポリオキシエチレンアルキルエーテル、ポリオキシエチレンアルキルフェニルエーテル、エチレングリコールモノ脂肪酸エステル、プロピレングリコールモノ脂肪酸エステル、ソルビタンモノ脂肪酸エステル、ソルビタントリ脂肪酸エステル、ポリオキシエチレンソルビタントリ脂肪酸エステル、ポリオキシエチレンモノ脂肪酸エステル、ポリオキシエチレンジ脂肪酸エステル、ポリオキシエチレン



アロピレングリコール脂肪酸エステルあるいはPOE多価アルコール類などが例示され、1種または2種以上を併用してもよい。用いる乳化剤のHLB値は8～14のものが好ましく、2種以上併用することがより好ましい。(C)成分の配合量は、(B)成分の粘性により大幅に減量が可能で、(A)成分における(a)、(b)および(c)の合計量100重量部に対して0.1～10重量部、好ましくは0.5～7重量部である。0.1重量部より少ないと安定なシリコンゴムスポンジ用エマルジョンとはなり難く、10重量部を超えるとシリコンゴムスポンジの耐熱後の物理特性が著しく低下するので、いずれも好ましくない。

#### 【0020】

かかる(A)成分、(B)成分、(C)成分から成るシリコンゴムスポンジ用エマルジョンは、例えば次のような方法で製造することができる。所定量の(A)液状シリコンゴム組成物と所定量の(B)水溶性ポリマーを含有する水と所定量の(C)乳化剤を高速ミキサーに投入し、所定時間混合するか、あるいは(A)成分中の(a)成分、(B)水溶性高分子を含有する水、(C)乳化剤の各所定量を高速ミキサーに投入し、所定時間混合した後、所定量の(A)成分中の(b)成分、(c)成分を混合してもよい。ここで使用するミキサーとしてはホモミキサー、パドルミキサー、ヘンシェルミキサー、ホモディスパー、コロイドミキサー、真空混合ミキサーあるいは2軸押出機等が例示されるが、安定なエマルジョンが得られるものであれば、特に限定されない。

#### 【0021】

かかるシリコンゴムスポンジ用エマルジョンが、混合時に気泡を巻き込んでいる時は脱気後、例えば、圧縮成形金型で、常温～120℃、好ましくは50～100℃の温度で硬化して湿潤状態のシリコンゴムスポンジ様成形体を形成させ、次に150～250℃の2次加熱処理で湿潤状態のシリコンゴムスポンジ成形体から水を除去することにより、セルが均一でしかも細かく、寸法精度のよいシリコンゴムスポンジを得ることができる。また、シリコンゴム用エマルジョンをロッド状にノズルから押出して、例えば80～100℃の熱水中で硬化させ、それを熱風乾燥して紐状のシリコンゴムスポンジを作製することもできる。

#### 【0022】

##### 【実施例】

以下、本発明を実施例、比較例により詳細に説明する。以下の記載において、部とあるのは重量部を、%とあるのは重量%をそれぞれ意味し、また粘度は25℃での値を示す。シリコンゴムスポンジ用エマルジョンならびにシリコンゴムスポンジの各特性は下記の条件で測定した。

- ・乳化状態： 所定の時間混合後、目視により外観で乳化の状態を判定した。
- ・スポンジの密度： シリコンゴムスポンジ用エマルジョンをシート状に成形硬化した後、熱風乾燥し、得られた成形シートを一定直径のポンチで打ち抜き、その打ち抜いたゴム片の重量と厚みを測定し、重量/体積( $g/cm^3$ )を密度として算出した。
- ・寸法精度： 熱風乾燥後の成形シート厚さ(mm)を熱風乾燥前の硬化成形シート厚さ(mm)で割った寸法比を寸法精度として算出した。
- ・セル径： 拡大鏡により観察した。

#### 【0028】

##### 実施例1～8

分子鎖両末端がジメチルビニルシロキシ基で封鎖された粘度10,000mPa・Sのジメチルポリシロキサン(ビニル基含有量0.14%)[(a)成分]100部、比表面積200m<sup>2</sup>/gのヒュームドシリカ20部、シリカ処理剤としてヘキサメチルジシラザン5部、水1部を加えて混合機で均一に混合し、180℃、減圧下で2時間熱処理を行い、流動性のある液状シリコンゴムベースを調製した。このベース100部に、両末端トリメチルシロキシ基封鎖ジメチルシロキサン/メチルハイドロジェンシロキサン共重合体(ケイ素原子結合水素含有量0.8%)[(b)成分]0.6部と塩化白金酸とジビニルテ

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トラメチルジシロキサンとの白金錯体〔(c)成分〕0.1部(白金濃度0.4%)と反応抑制剤として8,6-ジメチル-1-ヘキシル-3-オール0.1部を加えて均一に混合して液状シリコンゴム組成物を得た。この液状シリコンゴム組成物に水溶性ポリマーであるポリアクリル酸ソーダを含有する水(水溶性ポリマー含有量1.0%)、非イオン界面活性剤としてHLB値8.6のポリオキシエチレンジラウレート、HLB値10.4のポリオキシエチレンジオレエートを表1に示す配合比〔前記液状シリコンゴム組成物中の(a)、(b)および(c)成分の合計量100部に対する配合比〕で、市販のアビテックスミキサー(株)マツバラ社製)に投入し、回転数9000rpmで10分間混合して、それぞれシリコンゴムスポンジ用エマルジョンを調製した。

【0024】

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次に、これら気泡混入のシリコンゴムスポンジ用エマルジョンから気泡を除くため真空ポンプで脱気した後、厚さ2mmのシート状金型に流し込み、90℃、10分間かけて硬化し、湿潤状態のシリコンゴムスポンジ様成形シートを得た。これら成形シートを150℃の熱風式オーブンに入れ、1時間かけて熱風乾燥し、それぞれシート状のシリコンゴムスポンジを得た。その密度、寸法精度およびセル径を測定し、表1に示した。この結果から、本発明によるシリコンゴムスポンジはセルの均一性もよく、寸法精度に優れるものであった。

【0025】

比較例1～2

水溶性ポリマーを含まない以外はすべて実施例1、2の手順に従い、表1の配合比でシリコンゴムスポンジ用エマルジョンの調製を行ったが、安定なエマルジョンが作製できず、湿潤状態のシリコンゴムスポンジ様成形体を得ることができなかった。

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比較例3

実施例1～3の液状シリコンゴム組成物100部、吸水性ポリマー(三洋化成工業社製のアクリル酸塩重合体部分ナトリウム塩架橋物)を含有する水(吸水性ポリマー2.0%)120部、ポリオキシエチレンジラウレート7部を加えて混合し、シリコンゴムスポンジ用エマルジョンを調製し、実施例1～3の方法で硬化、熱風乾燥し、シリコンゴムスポンジを作製した結果を表1に示した。

【0026】

【表1】

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	実施例1	実施例2	実施例3	比較例1	比較例2	比較例3
液状シリコーンゴム組成物 (部)(*)	100	100	100	100	100	100
水溶性ポリマー(1.0%)を 含有する水(部)	120	180	120			
吸水性ポリマー(2.0%)を 含有する水(部)						120
水(部)				60	60	
ポリオキシエチレンジオレエ ート(部)	0.6	0.6	0.6		12	
ポリオキシエチレンジラウレ ート(部)	2.5	2.5		18		7
乳化状態	乳化	乳化	乳化	乳化せず	乳化せず	乳化
密度	0.58	0.48	0.61			0.57
寸法精度	1.0	1.0	0.99			0.99
セル径(mm)	0.1~0.2	0.1~0.2	0.1以下			0.2~0.8

(\*) (a), (b) および (c) の合計量 100 部

#### 【0027】

##### 実施例4

分子鎖両末端がジメチルビニルシロキシ基で封鎖された粘度40,000 mPa・Sのメチルビニルポリシロキサンポリマー(ビニル基含有量0.14%)[(a)成分]100部に、ジメチルジクロロシランで表面処理したアエロジルR-972(日本アエロジル社製)20部を加えて均一に混合し、180℃で2時間熱処理を行って、流動性のある液状シリコーンペースを調製した。このペース100部に、両末端トリメチルシロキシ基封鎖ジメチルシロキサン/メチルヒドロジェンシロキサン共重合体(ケイ素原子結合水素含有量0.8%)[(b)成分]0.5部と塩化白金酸とジビニルテトラメチルジシロキサンとの白金錯体[(c)成分]0.1部(白金濃度0.4%)と反応抑止剤として8,5-ジメチル-1-ヘキシル-3-オール0.1部を加えて均一に混合して液状シリコーンゴム組成物を得た。この液状シリコーンゴム組成物に前記の(a)、(b)および(c)の合計量100部に対する配合比として、水溶性ポリマーであるポリアクリル酸ソーダを含有する水(水溶性ポリマー含有量2%)120部、高級アルコール系非イオン界面活性剤としてHLB10.5のサンノックS8-50(三洋化成工業社製)2.5部、HLB12.8のサンノックS8-70(三洋化成工業社製)2.5部を容器に計量し、T.K.ホモミクサーMARKII2.5型(特殊機化工業社製)を使用して、回転数5000rpmで5分間かけて混合し、シリコーンゴムスポンジ用エマルジョンを調製した。

#### 【0028】

このシリコーンゴムスポンジ用エマルジョンを真空ポンプで脱気した後、厚さ2mmのシート状金型に流し込み、90℃で15分かけて硬化し、湿潤状態のシリコーンゴムスポンジ模成形シートを得た。これを成形シートを実施例1と同様な方法で熱風乾燥し、シリコーンゴムスポンジを得た。その密度、寸法精度およびセル径を測定した。その結果、密

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度は0.59、寸法精度は0.98、セル径は0.05~0.1mmであった。

【0029】

比較例4

実施例4において、水溶性ポリマーの替わりに吸水性ポリマーとしてアクリル酸塩重合体部分ナトリウム塩架橋物でゲル化させた水（吸水性ポリマー含有量0.3%）120部を使用する以外は全て実施例4と同条件で実施した結果、密度は0.58、寸法精度は0.98、セル径は0.2~1.0mmであった。

実施例5

沈降シリカ系の液状シリコンゴム組成物DY85-700A/B（東レ・ダウコーニング・シリコン社製）100部に、水溶性ポリマーとしてポリアクリル酸ソーダを含有する水（水溶性ポリマー含有量2%）120部、高級アルコール系非イオン界面活性剤としてサンノック88-50 2.5部、サンノック88-70 2.5部を容器に計量し、T.K.ホモミキサーを使用し、回転数5000rpmで5分間かけて混合し、シリコンゴム用エマルジョンを調製した後、実施例4と同様な方法で、脱気、硬化、熱風乾燥し、シリコンゴムスポンジを得た。測定の結果、密度は0.99、寸法精度は0.99、セル径は0.1mm以下であった。

【0080】

【発明の効果】

本発明のシリコンゴムスポンジ用エマルジョン組成物は（A）成分～（C）成分を含み、エマルジョンの状態では硬化して、湿潤状態のシリコンゴムスポンジ様成形体を形成できるといふ特徴を有する。また本発明のシリコンゴムスポンジの製造方法は湿潤状態のシリコンゴムスポンジ様成形体を熱風乾燥してシリコンゴムスポンジとなるので、金型形状の追従性も良く、かつ寸法精度も良好で、セルの均一性に優れ、しかもセルの細かいシリコンゴムスポンジを効率よく製造できるといふ特徴を有する。

【手続補正書】

【提出日】平成16年1月28日(2004.1.28)

【手続補正1】

【補正対象書類名】明細書

【補正対象項目名】0027

【補正方法】変更

【補正の内容】

【0027】

実施例4

分子鎖両末端がジメチルビニルシロキシ基で封鎖された粘度40.000mPa・sのメチルビニルポリシロキサンポリマー（ビニル基含有量0.14%）〔（a）成分〕100部に、ジメチルジクロロシランで表面処理したアエロジルR-972（日本アエロジル社製）20部を加えて均一に混合し、180℃で2時間熱処理を行って、流動性のある液状シリコンペースを調製した。このペース100部に、両末端トリメチルシロキシ基封鎖ジメチルシロキサン/メチルハイドロジェンシロキサン共重合体（ケイ素原子結合水素含有量0.8%）〔（b）成分〕0.5部と塩化白金酸とジビニルテトラメチルジシロキサンとの白金錯体〔（c）成分〕0.1部（白金濃度0.4%）と反応抑制剤として3,5-ジメチル-1-ヘキシル-8オール0.1部を加えて均一に混合して液状シリコンゴム組成物を得た。この液状シリコンゴム組成物に前記の（a）、（b）および（c）の合計量100部に対する配合比として、水溶性ポリマーであるポリアクリル酸ソーダを含有する水（水溶性ポリマー含有量2%）120部、高級アルコール系非イオン界面活性剤としてHLB10.5のサンノック88-50（三洋化成工業社製）2.5部、HLB12.8のサンノック88-70（三洋化成工業社製）2.5部を容器に計量し、T.K.ホモミキサーMARKII 2.5型（特殊機化工業社製）を使用して、回転数5000rpmで5分間かけて混合し、シリコンゴムスポンジ用エマルジョンを調製した。

【手続補正2】

【補正対象書類名】明細書

【補正対象項目名】0029

【補正方法】変更

【補正の内容】

【0029】

比較例4

実施例4において、水溶性ポリマーの替わりに吸水性ポリマーとしてアクリル酸塩重合体部分ナトリウム塩架橋物でゲル化させた水（吸水性ポリマー含有量0.3%）120部を使用する以外は全て実施例4と同条件で実施した結果、密度は0.58、寸法精度は0.98、セル径は0.2～1.0mmであった。

実施例5

沈降シリカ系の液状シリコーンゴム組成物DY85-700A/B（東レ・ダウコーニング・シリコーン社製）100部に、水溶性ポリマーとしてポリアクリル酸ソーダを含有する水（水溶性ポリマー含有量2%）120部、高級アルコール系非イオン界面活性剤としてサンノックSS-50 2.5部、サンノックSS-70 2.5部を容器に計量し、T.K.ホモミキサーを使用し、回転数5000rpmで5分間かけて混合し、シリコーンゴム用エマルジョンを調製した後、実施例4と同様な方法で、脱気、硬化、熱風乾燥し、シリコーンゴムスポンジを得た。測定の結果、密度は0.59、寸法精度は0.99、セル径は0.1mm以下であった。

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フロントページの続き

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